AXAA e-newsletter

Issue 2, 2016 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

Organisation of AXAA-2017 is well and truly in full swing, with our line-up of exciting Plenary presentations finalised, as well as a developing and diverse line-up of Invited speakers from across Australia and internationally, presenting on a range of topics and techniques. We are also excited about our Public Lecture, which will quite literally be out of this world! Please visit the Speakers page of the Conference website for more details.

The Workshops component of AXAA-2017 has also taken shape, with our XRD and XRF Basics workshops for new practitioners running alongside an Advanced workshop stream featuring sessions on tomography, pair distribution function analysis, thin film analysis, and complementary analysis techniques.

A new addition to AXAA-2017 will be a career path panel discussion session, featuring panellists with varied backgrounds, career progression and experience. This promises to be a valuable session for students and people in the early stages of their careers, especially.

So now is the time to be submitting abstracts (abstract submission deadline is 7th October 2016) and registering (early bird registration closes 18th November 2016) for what's going to be a fantastic event.

If you have an interest in holding a pre or post-conference tour now is the time to contact us. Similarly, if you would like to be involved in AXAA-2017 as a Sponsor, please contact us or check out the sponsorship prospectus on the website.

Student bursaries are available for AXAA-2017, through our AXAA Student Seminars events to be held in NSW, SA, QLD, VIC and WA in September and October. You probably would have seen these advertised separately through our mailing list. These are high quality events showcasing young talent in X-ray/neutron analysis. Please contact us if you are interested in sponsoring a Student Seminars event.

In this Newsletter we are excited to announce "A Day in the Life of an X-ray Scientist", which is a new series where we get to know members of the AXAA community. We have had two profiles so far (Joel O'Dwyer from CSIRO, and Marek Rouillon from Macquarie University), and are seeking new entries so if you would like to participate, or nominate someone, please contact us. Our thanks to AXAA member Jessica Hamilton (Monash University) for getting this new series going!

Now is the time to consider nominations for the Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis, the Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis, and the PANalytical Award for

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Excellence in Analysis by an Early Career Scientist. These prestigious will be awarded at AXAA-2017. Please read on for details of the nomination and selection processes.

Finally, now is also the time to be thinking about nominations for members of the next AXAA National Council, which will be elected in the AGM at AXAA-2017. This is your chance to be intimately involved in the organisation and direction setting of AXAA, and the greater number of dynamic and enthusiastic people involved, the better to ensure a vibrant and diverse community.

Nathan Webster
AXAA President

Vale Dr Hugo Rietveld (1932 – 2016) - Developer of the Rietveld Refinement Method

AXAA members will be saddened to hear of the passing of Dr Hugo Rietveld in The Netherlands on 16 July 2016 after a short illness. He leaves behind his dear spouse Truus, a son and two daughters. Recent days have seen various tributes to Hugo's scientific achievements, notably that by Alan Hewat, Bill David and Lambert van Eijck at

https://www.mailarchive.com/rietveld_l@ill.fr/msg06023.html

This personal reflection by Brian O'Connor recalls a friendship of more than 50 years, going back to the 1960s when we were PhD students in the Crystallography Group at the University of Western Australia led by the late Dr Ted Maslen. These were especially exciting times in Australian crystallography with the availability of neutron diffraction using the brand new HIFAR research reactor at Lucas Heights. During Hugo's PhD studies, he co-pioneered single crystal neutron diffraction at Lucas Heights in a structural crystallography study which was published in Nature in 1961 [1]. The events surrounding this pioneering work are given in the history - *AINSE, An institute for research and training excellence in nuclear science - The First 50 Years. A history of the Australian Institute of Nuclear Science and Engineering (2008) Brian H O'Connor, Allan R Chivas, Dennis W Mather, John D C Studdert, Anna E Binnie* (pages 1,15) [2].

On completing his PhD, Hugo maintained a strong connection with his adopted Australia after returning to his native The Netherlands to join the neutron diffraction group of the *Reactor Centrum Nederland* in Petten where he focused on working with powders for which large single crystals were not available. This was most challenging due to overlapping reflections making crystal structure solution and refinement unduly difficult. Hugo moved quickly in developing what was to become the Rietveld Method, and presented a paper at the International Union of Crystallography Congress held in Moscow in 1966. He later confided to me that he was deeply disappointed that not even one question was asked at the end of his talk. Hugo then published his two epic papers on the method, notably "A profile refinement method for nuclear and magnetic structures" [3].

Take-up of the method was relatively slow initially, with there being 191 journal citations up to 1977 when it was realised by X-ray diffractionists that the method would be equally useful for X-ray powder data. The growth in citations has been phenomenal – in 2016 the number of citations will be in the vicinity of 1,000 and the annual citation number is still increasing rapidly. To date, there have been almost 15,000 citations. As the method uptake exploded through X-ray diffraction usage, it became known in 1978 as the Rietveld Method following a suggestion by the late Terry Sabine.

What Hugo might have achieved had he remained active in crystallography defies imagination. Shortly after publishing his extraordinary paper, and disappointed by the lack of funding available for neutron diffraction, he became head of the library department at Petten and remained in administration for the remainder of his career. Eventually, Hugo received many prestigious awards for his work, perhaps the most significant being the 1995 Gregori Aminoff Prize for Crystallography conferred by the Royal Swedish Academy of Sciences. Figure 1 shows Hugo receiving the award from the King of Sweden at the presentation ceremony in Stockholm.

Figure 1. Hugo Rietveld receives the *Gregori Aminoff Prize for Crystallography* from the King of Sweden in 1995.

For all of his scientific achievements, Hugo was a modest and very caring person and most generous with his time to those interested in his work. X-ray scientists in Western Australia were most fortunate that Hugo and Truus made various visits back to Perth to see family. We were very proud that Hugo agreed to be the guest-of-honour at the International Union of Crystallography International

Symposium on X-ray Powder Diffractometry held in Fremantle 20-23 August, 1987. This gathering of the world who's who of powder diffraction was organised by Brian O'Connor (Curtin), the late Dr Jim Graham and the late Dr Ernie Nickel (CSIRO), and Dr Tony Bagshaw (Alcoa). The proceedings are available at Australian Journal of Physics (1988, vol. 41 no. 2) [4]. Many of those luminaries present met Hugo for the first time which was thrilling for them and even more so for Hugo. Figure 2 shows Hugo at the Symposium dinner.

Figure 2. Hugo Rietveld at IUCr Symposium dinner at the Esplanade Hotel, Fremantle in 1987. From left – the late Prof Deane K Smith (Pennsylvania State University), Hugo, and Brian and Jennifer O'Connor.

During the 1980s, the Rietveld method was being developed for phase composition analysis, with the first papers on the method appearing in 1987-88. It was first described for use in neutron powder diffraction [5], and shortly after two papers on XRD methodology appeared [6,7]. At the time, Hugo appeared to be only moderately interested in phase analysis applications, and was politely bemused by my comments whenever we met that applications of his method in industrial materials characterisation would ultimately stand alongside its long-standing use in fundamental research.

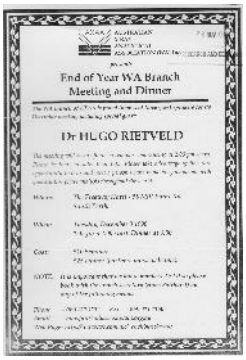

Figure 3. Hugo Rietveld at AXAA Annual Meeting/Dinner in Perth, 1996. Hugo with Hans Fairhurst (Cockburn Cement) – Branch Chairman – and Sandra Fairhurst.

The WA Branch of AXAA was most fortunate that Hugo was guest speaker at the Branch end-of-year meeting in 1998 – see Figure 3. By then some XRD powder analysts in the resources sector were starting to see the potential value of Rietveld phase composition analysis in the industrial setting. Hugo gained a better appreciation of the potential through talking to AXAA members and also on visits to Curtin University where he met many research staff and students who were routinely using his Rietveld analysis for materials characterisation – see Figure 4. Almost 20 years after that important AXAA meeting, the crucial importance of the technique in process control in mining and mineral processing is starting to be accepted. But is has been a slow journey with some still trying to do 'semi-quantitative' analysis using the peak ratio technique. One day ….

Figure 4. Hugo Rietveld meets students and staff using of his method for materials characterization, during a visit to Curtin University in 1996. Included in the photograph are Dr Husin Sitepu (now at Saudi Aramco), Dr John Carter (Bureau Veritas), Dr Phe Man Suherman (GSI, Germany), Dr Rob Skala (USA), Prof Suminar Pratapa and Prof Athanasius Bayuseno (Indonesia), and currently in Perth - Dr Bee Gan, Prof D Li and Prof Jim Low.

I join the legion of Rietveld practitioners in celebrating the life of this wonderful person as well his scientific legacy. Our thoughts are especially with Truus Rietveld and family at this very sad time.

Brian O'Connor
Curtin University

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Preferred Orientation in Non-Bragg-Brentano Geometries

With the growth in synchrotron experimentation, the use of *in situ* techniques, and the availability of multiple optical configurations for laboratory instruments, more measurements are being conducted in geometries that deviate from the de facto Bragg-Brentano standard. As the experimental geometry changes, so too does the way in which preferred orientation manifests itself in the diffraction pattern. An understanding of the data collection geometry allows for an understanding of how this, and other, factors differ, and how to correct their effects in Rietveld [1] modelling.

The March-Dollase correction for preferred orientation [2] is given by:

$$P_{MD} = \left(r^2\cos^2\rho + \frac{\sin^2\rho}{r}\right)^{-\frac{2}{3}}$$

where ρ is the angle between the preferred orientation direction (**p***) and the specimen normal (**n***), which is taken to be the direction of the specimen's rotational symmetry, and r is a measure of the degree of orientation, where r = 1 is a random powder. In symmetric reflection geometry, the specimen normal and the diffraction vector (**d***) are parallel, which allows ρ to be given simply as α, the angle between **d*** and **p***, simplifying the calculations – see Fig. 1(a).

If the symmetric reflection implementation of the March-Dollase preferred orientation correction is applied directly to data collected in Debye-Scherrer geometry, this results in r being approximated as $r^{-1/2}$, but only for values close to unity [3]. As the powder becomes progressively more oriented, the correction becomes significantly worse. The Debye-Scherrer geometry, with respect to preferred orientation corrections, is shown in Fig. 1(b), where the specimen normal is at right angles to the diffraction vector. Application of the March-Dollase correction, as is, to asymmetric reflection is totally incorrect, as the angles between **d***, **n***, and **p*** are continuously changing – see Fig. 1(c). The relevant geometry for symmetric and asymmetric transmission is shown in Figs. 1(d) & 1(e) – it can be seen how the geometry is similar to that of Debye-Scherrer and asymmetric reflection.

Ida [4] comprehensively covers the application of the March-Dollase model to asymmetric reflection and Debye-Scherrer geometries, and shows that a simple implementation of the preferred orientation correction, f, can be given by

$$f(r,\alpha,\Delta) = \frac{1}{2\pi}\int_0^{2\pi} g(r,\alpha,\Delta,\varphi)d\varphi$$

$$g(r,\alpha,\Delta,\varphi) = P_{MD}$$

$$\cos\rho = \cos\alpha\cos\Delta - \sin\alpha\sin\Delta\sin\varphi$$

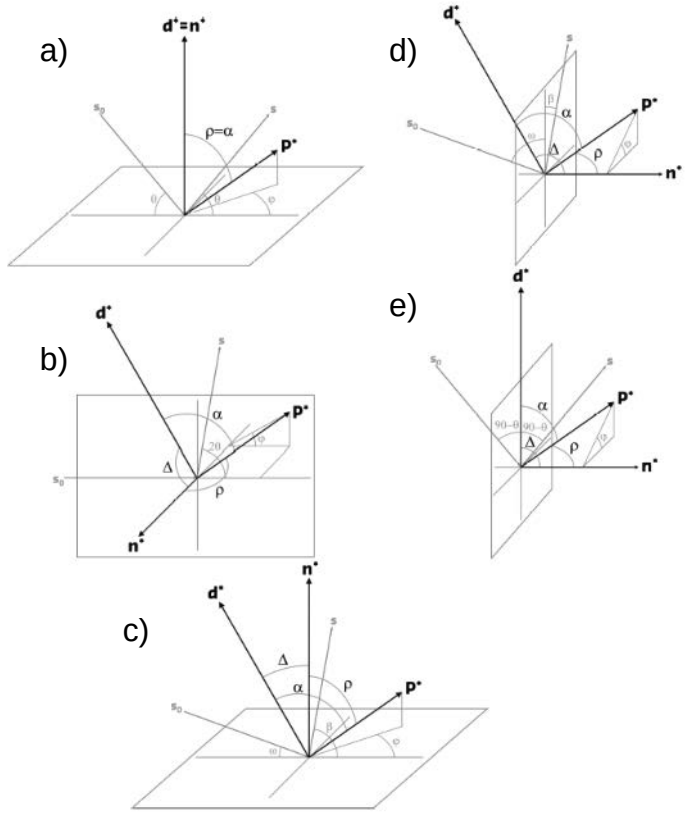

Figure 1. Required vectors and angles for both March-Dollase and spherical harmonics corrections for (a) symmetric reflection, (b) Debye-Scherrer, (c) asymmetric reflection, (d) asymmetric transmission, and (e) symmetric transmission geometries. **d*** is the diffraction vector, **n*** is the rotation axis of the specimen, and **p*** is the preferred orientation vector. The angle between **d*** & **n*** is Δ, **d*** & **p*** is α, and **n*** & **p*** is ρ. ϕ is the rotation angle about **n*** of the projection of **p*** onto the plane defined by **n***. $\mathbf{s_0}$ and **s** are the incident and diffracted beams, respectively.

where ρ is the angle between **n*** & **p***, α is the angle between the **d*** & **p***, ϕ is the angle between **d*** & **n***, and Δ is the rotation angle about **n*** of the projection of **p*** onto the plane defined by **n*** – see Fig. 1. In the simplest case, the integral can be replaced by a sum

$$f(r,\alpha,\Delta) \approx \frac{1}{N}\sum_{j=0}^{N-1} g(r,\alpha,\Delta,\varphi)$$

where N = 16 is appropriate for most applications. If r >> 1, this summation approach breaks down, and the other, more rigorous approaches outlined by Ida should be applied. However, if r moves too far from unity, then the March-Dollase correction itself is also probably not an appropriate model [4].

The same alteration is also relevant to the use of spherical harmonics [5], where the intensity correction is given by

$$\bar{W}(hkl,\Delta) = \sum_{ij} C_{ij}Y_{ij}(\theta_{hkl},\varphi_{hkl})\,P_i(\cos\Delta)$$

where C_{ij} are adjustable parameters, Y_{ij} are the symmetrised spherical harmonics, Δ is the angle between the **d*** and **n***, and $P_i(x)$ is the i^{th}-order Legendre polynomial. In symmetric reflection, Δ = 0°, and all of the

associated Legendre polynomials have a value 1, and as such, don't influence the correction. In Debye-Scherrer, asymmetric reflection, asymmetric transmission, and symmetric transmission geometries, Δ has values of 90°, θ - ω, θ + ω, and 90°, respectively, and so these angles must be included in the Legendre polynomials in order to obtain representative corrections.

The corrections outlined in this note have been implemented in the TOPAS (Bruker AXS, 2014) macro language and are available on the TOPAS wiki [6,7].

As with any preferred orientation corrections, it is almost always better to carry out a texture analysis to properly quantify the orientation present in the specimen [8], but this is not often appropriate for in situ studies. With these corrections, the approximate model is being applied exactly correctly in the new geometries, which discounts any effect an incorrect implementation may have on any analysis.

Matthew R. Rowles
Department of Physics and Astronomy, Curtin University

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International Conference on Li Batteries Wrap-Up

The International Meeting on Lithium Batteries is one of the, if not the biggest conference for battery research. The 18th meeting was held in Chicago, Illinois, United States from 19-24th June 2016 consisting of 75 plenary talks by the leaders in the field with more than 1000 posters from industry, universities, and research institutions. The topics were not limited to lithium batteries only, other battery systems such as Na, Mg, Ca, Li/Na-S, and even recycling of batteries were thoroughly discussed.

X-ray based techniques have always been a crucial tool used by battery researchers to characterize materials inside their batteries. Ranging from X-ray diffraction, absorption spectroscopy to microscopy and tomography, X-ray methods are used by researchers to obtain specific information about material function inside batteries. This meeting in particular featured a significant number of talks involving *in situ* and *in operando* X-ray techniques (and neutron techniques). This highlights the evolving role of X-ray techniques in future research, in the battery field the move towards more and more *in situ* and *operando* studies. There is a clear need for researchers to see how their material behaves in real applications.

Strong student participation is also seen as there are more than 600 PhD students that attended this meeting. Over the course of 5 days, participants engaged in networking through the poster sessions which spanned over 3 nights and the gala dinner.

Attending one of the best conferences for the battery research community is an exciting experience. Being able to see and hear all the leaders from the field in person and meet other researchers that share similar goals can be both an encouraging and overwhelming. With a vast number of students and academics, the conference is an excellent venue to make new connections and collaborations.

James Christian Pramudita
PhD Candidate, School of Chemistry, UNSW

AXAA Awards for Excellence in X-ray Analysis: Call for Nominations

Closing Date for Submission of Nominations:
28 October 2016

The Awards

These two awards, one for XRF and one for XRD, are for "significant long term contributions" to X-ray analysis rather than say a single paper, and will perpetuate the contribution of the person after whom the award is named.

XRF – Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis

XRD – Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis

Form of Awards
Each award will comprise an engraved medal.

Selection Criteria

1. The principal criterion will be the excellence of the applicant's development of high-impact, innovative X-ray analysis methods and their take-up by the X-ray analysis community. Work in which XRF or XRD has been a peripheral tool will not be considered.

2. The period over which the contribution is to be considered will be at least 5 years.
3. All or most of the cited work will have been conducted in Australia.
4. The recipient will have been a member of AXAA for at least 5 years prior to the application being submitted.
5. It is desirable, but not essential, that the applicant has contributed to AXAA in a substantial way, for example through quality presentations at AXAA national conferences and/or administrative service for AXAA.
6. Past recipients of an AXAA XRD or XRD award will not be considered for a second award in the same category.

Applications

Applications will be submitted by a nominator on behalf of the applicant. The documentation will comprise:

- CV.
- Publication list. This may include items protected by confidentiality if the applicant can pre-arrange an appropriate confidentiality agreement.
- Advocacy statement highlighting the application's alignment with the selection criteria.
- Names and contacts for three technical referees, one being the nominator.

Applications are to be submitted in Nathan Webster (Nathan.Webster@csiro.au). Please send to Nathan as an attachment.

The PANalytical Award for Excellence in Analysis by an Early Career Scientist

Closing Date for Submission of Nominations:
28 October 2016

This PANalytical-sponsored award serves to encourage the development of dynamic, young analysts in one of the various fields of analytical science of interest to the Australian X-ray Analytical Association (AXAA) membership. The award will celebrate "outstanding application of laboratory and/or major radiation facility analytical technique(s) so as to achieve significant impact in a field of endeavour". The recipient of the award will not be limited to having used any specific brand of instrument.

The award will consist of $2000 to be used for professional development.

Selection Criteria:

1. The principal criterion is excellence of the application of analytical technique(s) so as to achieve significant impact in the field of endeavour. Work in areas beyond those central to AXAA's activities (e.g. X-rays, neutrons, minerals, materials, processing, environment, energy, cultural heritage) will not be considered.
2. The recipient will be an Early Career Scientist (less than 5 years in industry or since PhD conferral at the time of the AXAA-2017 conference).

Applications:

Applications submitted by either a nominator or the nominee will be accepted and will comprise:

1. The nominee's CV (including a publication list).
2. A detailed citation (statement, no more than three pages) highlighting the excellence of the nominee's contribution and addressing the primary selection criterion.

Applications are to be submitted in electronic form to the AXAA President, Nathan Webster.
Nathan.Webster@csiro.au

Selection Process:

1. The AXAA National Council will appoint a selection panel of three persons for the award.
2. The selection panel will make a recommendation to the AXAA National Council who, along with PANalytical, will make a formal decision on the recommendation. The decision will be final, and there will be no appeal process.
3. The AXAA National Council reserves the right to withhold the award if no suitable candidates of sufficiently high standard are nominated.

The award will be presented at the AXAA 2017 Schools, Conference and Exhibition (AXAA-2017).

AXAA National Council, 2017-2020

In accordance with the requirements of the AXAA-Inc constitution, the current National Council will vacate their positions at the next AGM, to be held during the AXAA-2017 conference.

We seek nominations for the 2017-2020 National Council, comprising six members, with three being nominated by the retiring Council and three being nominated by the AXAA members.

We request that AXAA members forward their nominations for any National Council position, including General Council, President, Vice-President, Secretary, Treasurer and Newsletter Editor positions, to the AXAA Secretary Natasha Wright (Natasha.Wright@csiro.au).

Closing Date for Nominations: 6th January 2017

Upcoming Events

RACI Materials Chemistry one-day symposium

22 August 2016
UNSW Sydney

The RACI Materials Chemistry one-day symposium will bring together researchers with a vested interested in designing and making better Materials! The symposium will feature student presentations and talks from international delegates. The symposium will start at 9:45 for registration and run through to 6pm with an optional dinner in the evening.

For more information:
Email: Neeraj.Sharma@unsw.edu.au
Website: https://www.raci.org.au/events/category/materials-chemistry-division

AXAA Website and Contacts

Please visit our website, www.axaa.org, for further information, or follow us on Twitter @axaa_org.

NATIONAL COUNCIL PRESIDENT:
Nathan Webster
CSIRO Minerals Resources, Box 10, Clayton South, VIC 3169
Telephone: (03) 9545 8635
e-mail: nathan.webster@csiro.au

NATIONAL COUNCIL VICE PRESIDENT:
Vanessa Peterson
Bragg Institute, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 9401
e-mail: vanessa.peterson@ansto.gov.au

NATIONAL COUNCIL SECRETARY
Natasha Wright
CSIRO Manufacturing, Normanby Rd, Clayton, VIC 3168
Telephone: (03) 9545 2041
e-mail: natasha.wright@csiro.au

NATIONAL COUNCIL TREASURER
Gordon Thorogood
Institute of Materials and Engineering, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 3183
e-mail: gordon.thorogood@ansto.gov.au

NATIONAL COUNCIL NEWSLETTER EDITOR
Mark Styles
CSIRO Manufacturing, Private Bag 10, Clayton South, VIC 3169
Telephone: (03) 9545 8179
e-mail: mark.styles@csiro.au

Please email contributions for Issue 3 of the 2016 AXAA Newsletter to Mark Styles by Friday the 25th of November. Any comments or feedback about the Newsletter are welcome.

AXAA Membership

All registered participants of the AXAA-2014 conference are automatically granted AXAA membership for 3 years. Alternatively, new memberships can be obtained free of charge, by making an application to the National Council. Candidates should provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Natasha Wright (see AXAA contacts) if you would like to apply.

Bruker Australia / NZ News Jul 2016

In this update:

- The New D8 ADVANCE
- AMICS software acquisition

The New D8 ADVANCE - a plus for your research

Bruker AXS proudly presents the newest version of the D8 ADVANCE X-ray diffractometer. Thanks to the consistent implementation of our DAVINCI Design, new cutting edge technology has never been so easy to use.

The newly released **TRIO** optic uniquely enables completely automated switching between the three most commonly used primary beam geometries: divergent beam for conventional powder diffraction (XRPD), high intensity parallel beam for non-ideal powders, thin films, and micro-diffraction, and pure **Cu-Kα1 parallel beam** for high-resolution diffraction (HRXRD) of epitaxial thin films. As is the case for our established **TWIN** optic, switching between these geometries is done with push-button software control without any need for post-switching alignment. This saves time, increases accuracy, and lets the user concentrate on results instead of bothering with instrumentation.

The new D8 ADVANCE offers extended **XRD²** functionality. The fully-integrated **PILATUS3 100K-A 2D** detector from DECTRIS enables high-speed, enhanced micro-diffraction analysis. Noise-free data acquisition and large angular coverage in Gamma and 2Theta facilitate detecting extremely weak diffraction signals that often result from investigations on micrometer-size areas.

The extension of the analytical capabilities is complemented by smart solutions for sample mounting with the new compact **UMC** stage or the **Compact Cradle**^{plus}, which offers motorized z-translation, versatile sample mounting accessories, and automated height adjustment with the double-laser sample positioning system.

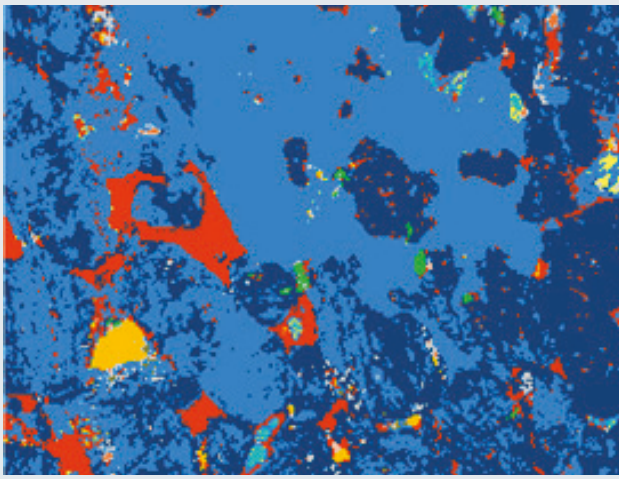
The new D8 ADVANCE – easy switching between all XRD applications.

Acquisition for AMICS for Advanced Minerals Identification and Characterization

Bruker announced that it has acquired the assets of Yingsheng Technology Pty Ltd. in Brisbane, Australia, that relate to the 3rd generation Advanced Mineral Identification and Characterization System (AMICS) software package, and respective IP for advanced minerals identification and characterization.

AMICS is a highly innovative software platform for automated identification and quantification of mineral and synthetic phases. It can be used in conjunction with either scanning electron microscopes (SEM) and SEM-related energy dispersive X-ray spectrometers (EDS), or now also with novel micro X-ray fluorescence (µXRF) analyzers. Its versatile, 3rd-generation automation and forward-thinking quantitative analysis capabilities make AMICS an ideal tool for research in geological and material sciences, as well as for fast, efficient and precise phase analysis in various industrial and exploration applications, such as mining, oil & gas, coal, cement, refining and recycling.

"We are very pleased with the addition of the AMICS software to our portfolio of analytical tools for electron microscopy and µXRF analysis," commented Thomas Schuelein, President of Bruker's Nano Analytics Division. "Making AMICS an integral part of our QUANTAX™ EDS systems will enable us to offer researchers and OEM partners in earth and material sciences enhanced analytical power for minerals characterization. With AMICS we can now even better serve the increasing demand in the mining and other industries for fast, highly productive and precise automated phase analysis."



AMICS mineral phase map of an exploration drill core, acquired with the M4 TORNADO™ µXRF system

Mr. Schuelein continued: "Furthermore, combining AMICS with our novel and performance-leading µXRF system M4 TORNADO™, will augment today's workflows in the fields of minerals exploration and production, as it will allow micro-scale analysis on large samples, such as rocks, drill core sections, etc., without elaborate sample preparation.

We are pleased that, as part of the asset purchase agreement, the key software and applications engineers who created AMICS under the direction of Yingsheng's founder and CEO, Dr. Ying Gu, have transitioned to Bruker, enabling continuity in the further development and commercialization of AMICS going forward."

Contact for enquiries
D8 ADVANCE: Martin Duriska 0487 800 317 or martin.duriska@bruker.com
AMICS: Jens Bergmann 61 3 9474 7000 or Jens.Bergmann@bruker.com

Bruker Pty Ltd ABN: 86 002 561 328 | Tel: 1300-BRUKER (1300-278-537) | Email: sales.anz@bruker.com | Web: www.bruker.com

Melbourne Office:
1/28A Albert St Preston VIC 3072
PO Box 8432, Northland Centre VIC 3072
Ph: (03) 9474-7000
Fax: (03) 9474-7070

Sydney Office:
Unit 7, 163-167 McEvoy St, Alexandria NSW 2015
PO Box 202, Alexandria NSW 1435
Ph: (02) 9550-6422
Fax: (02) 9550-3687

Wellington Office:
Unit 6, 5 Bouverie St Lower Hutt 5012, NZ
PO Box 39-202, Lower Hutt 5045, NZ
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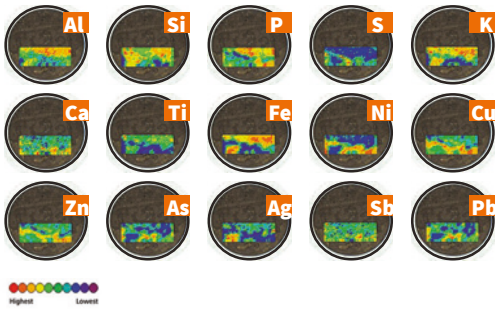

Figure 1: 2D contour plots showing the distribution and relative abundances for 15 elements detected in the below archeological coin

Qualitative application: Analysis of an archeological coin

In this study, a coin found in the region of Overijssel (the Netherlands) and dating back to the 19th century, was analyzed using qualitative intensity-based element mapping. Figure 1 shows the distribution and relative sensitivities of 15 elements within the analyzed area. Clear compositional differences are evident between the inner and outer part of the coin, suggesting that the coin surface was altered as a consequence of the interaction with the soil in which it was buried. The 2D images show that the substrate is enriched with Ni and Cu, while the corrosive outer layer is more enriched with Al, Si and Fe. Moreover, images show the presence of heavy metals in high concentrations (e.g. As and Pb), suggesting the inability of separating these elements during metal production. The elemental composition of the coin can help in reconstructing the origin of the metal used to forge it, as well as the provenance of the ores used in the metal smelting and can ultimately help identify trade routes.

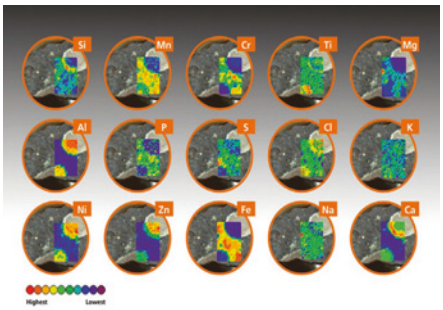

Figure 3: Relative concentrations of the elements

Spot No.	1	2	3	4	5	6
Compound	Concentration (%)	Concentration (%)	Concentration (%)	Concentration (%)	Concentration (%)	Concentration (%)
SiO_2	33.66	36.88	34.85	32.79	34.10	32.98
Al_2O_3	3.12	24.21	4.14	3.96	19.31	4.40
P_2O5	0.31	0.06	0.27	0.17	0.13	0.13
SO_3	0.48	0.41	0.34	0.44	0.62	0.51
Cl	0.01	0.36	0.06	0.09	0.10	0.03
NaO_2	0.43	2.88	0.48	0.15	2.40	0.21
MgO	25.82	12.56	34.51	24.85	17.92	24.60
K_2O	0.08	0.12	0.07	0.09	0.24	0.05
CaO	1.85	13.21	1.90	2.32	6.92	2.29
TiO_2	0.18	0.49	0.18	0.21	0.50	0.13
Cr_2O_3	0.66	0.22	0.58	0.59	0.29	0.49
Mn_2O_3	0.23	0.07	0.25	0.24	0.16	0.21
Fe_2O_3	32.67	7.50	22.05	33.52	15.87	33.52
NiO	0.47	0.94	0.32	0.56	1.39	0.44
ZnO	0.02	0.09	0.01	0.02	0.06	0.02
ZrO_2	0.004	0.004	0.003	0.004	0.01	0.004
Norm factor	1.02	1.04	0.97	1.01	1.02	0.99

Table 1: Quantitative results for the 6 spots measured

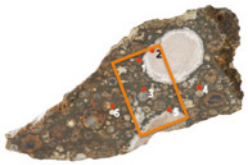

Figure 2: Measurement areas for the meteorite

Quantitative application: Compositional mapping of a meteoritic sample

In this study the distribution of a variety of elements was mapped in the North West Africa 2086 chondritic meteorite sample (type CV3), with calcium-aluminium-rich inclusions. Figure 3 shows the relative concentration of 15 elements from the analyzed area clearly demonstrating compositional differences between the host matrix of the sample and the Ca-Al-rich inclusions.

Table 1 shows the quantitative results for the 6 specific spots measured on the sample.

Measure up to 24,000 spots per sample with PANalytical's Zetium's small spot analysis and mapping module, which can accommodate samples with diameters up to 35mm. The measurement spots have a diameter of 500 µm (FWHM) and a minimum intervening step size of <100µm, which amounts to a maximum of 24,000 spots per sample.

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Since the launch of Zetium in 2015, it has received various accolades. In 2015, it was the finalist in the R&D100 awards, named one of top 3 products at Pittcon 2015. Most recently, it was recognised as the most outstanding new product at the prestigious Annual Conference of China Scientific Instruments in Beijing.

For more information on Zetium, visit www.panalytical.com/zetium

Read the full application note at PANalytical's Knowledge Centre

www.panalytical.com/Application-notes/Practical-small-spot-analysis-and-element-mapping.htm

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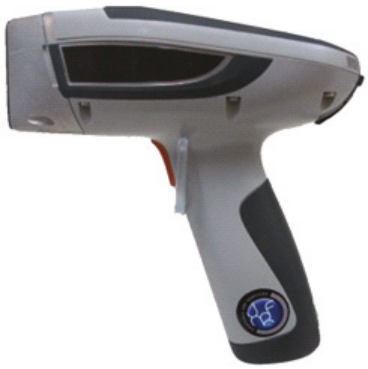
Itrax XRF Drillcore scanner versus a handheld XRF

Anders Rindby, PH. D. Cox Analytical Systems

Vs



Cox Analytical Systems has conducted a comparison between a handheld XRF unit, and the Itrax Drillcore scanner from Cox Analytical Systems. The handheld measurements were performed at the SP Technical Research Institute of Sweden.

Two different Standard Reference materials were used:
1. NIST SRM 1834, Fused Simulated Ore for XRF Fluorescence Spectrometry
2. USGS Green River Shale SGR-1 sediment standards

The SRM 1834 is a silica base glass disk which is supposed to be used as XRF standards for analysing rocks, ore and clay samples. The SGR-1 is a powder sediment standard from the Mahogany zone of the green River Formation which is a petroleum and carbonate-rich shale.

For the analysis with the handheld XRF, the two standards were analysed by a handheld instrument, provided and carried out by the SP Technical Research Institute of Sweden. The instrument was used in an s.c "mining" mode and each sample was exposed for 20 seconds in four different modes of tube voltage and tube anodes (in total 4x20, i.e. eighty seconds) to cover all elements from Al up to U.

The instrument was held in close proximity with the sample surface during measurements. The exposed area was estimated to be about 0.5 cm2.

The Itrax data was recorded from an Itrax Scanner where the standards were exposed for two seconds. The exposed area was about 0.012 cm2.

Table I & II shows the estimated concentrations recorded from the two XRF instruments together with the certified concentrations. The tables also show the estimated Detection Limits (D.L), in concentrations, for the two instruments.

The D.L.'s were estimated from the spectral peak/area ratio and calculated from the standard definitions given in XRF spectrometry (Handbook of X-Ray Spectroscopy" 2'nd edition, ed; R van Grieken, A.Markowicz, Marcel Dekker, Inc 2002).

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As seen from Table I & Table II the Itrax scanner provides somewhat more precise analytical data, especially for light elements (where the handheld instrument seems to give a systematic deviation) and also for heavy elements (Z>20) in low concentrations. For heavy elements in higher concentrations (> 2%) both instruments give reasonably good estimates.

It is also apparent from table I & II that the handheld instrument has lower detection limits for light elements like Al and Si, but for all heavier elements the Itrax is comparable or superior, and Itrax in total detects many more heavy elements (see tables).

Note that while the handheld instrument was recording an area of about 0.5 cm2 over 80 seconds the Itrax took only 2 seconds over an area of 0.012 cm2. This means that the Minimum Detectable Amount (at a fixed measuring time) for the Itrax will be several orders of magnitude lower than the handheld instrument for any given D.L in concentration.

In practical work, this difference can show up for example when detecting fine laminations. At the same time, the capacity of Itrax is much higher with a time for analysis of 1-2 seconds per point as compared to the 20 seconds needed with the handheld device.

The pros and cons of each instrument therefore need to be kept in mind for each specific application and desired usage.

IMP has proudly partnered with Cox Analytical Systems to offer their Drillcore scanner to the market.

Table I

Standard #1834	HandHeld XRF		Itrax Drillcore		
Element	Calculated concentration (%)	Detection limit (%)	Calculated concentration (%)	Detection limit (%)	Certified concentration (%)
Al	10.211	0.1400	21.6283	0.7523	20.700
Si	16.240	0.0430	23.1469	0.1934	20.200
P	0.178		Not detected	0.0601	
S	0.034		0.1176	0.0222	
Cl	Not Detected		0.0602	0.0096	
K	0.282		0.4357	0.0027	0.420
Ca	0.106		0.0997	0.0017	0.095
Ti	0.970	0.0045	1.1873	0.0009	1.110
V	0.009		Not detected	0.0007	
Cr	0.015		0.0237	0.0006	
Fe	0.308	0.0012	0.3477	0.0005	0.320
Co	Not detected		0.0024	0.0005	
Ni	Not detected		0.0028	0.0005	
Cu	Not detected		0.0023	0.0005	
Zn	Not detected		0.0071	0.0005	
Zr	0.044		0.0299	0.0004	
Ba	0.041		0.0623	0.0013	0.062

Table II

Standard SGR	HandHeld XRF		Itrax Drillcore		
Element	Calculated concentration (%)	Detection limit (%)	Calculated concentration (%)	Detection limit (%)	Certified concentration (%)
Al	1.098	0.22	3.0631	2.1724	3.450
Si	9.350	0.05	12.9398	0.8031	13.1700
P	0.071		0	0.2495	0.1430
S	1.223		1.4928	0.0932	1.5300
Cl	0.007		Not Detected	0.0411	
K	0.912		1.4901	0.0121	1.3700
Ca	5.098	0.009	6.0455	0.0079	5.9900
Ti	0.103		0.1393	0.0044	0.1510
V	0.011		0.0133	0.0036	0.0130
Cr	0.005		0.0051	0.0032	0.0030
Mn	Not detected		0.0297	0.0029	0.0267
Fe	1.417	0.0025	2.1630	0.0028	2.1180
Ni	Not detected		0.0084	0.0027	0.0029
Cu	Not detected		0.0106	0.0028	0.0066
Zn	0.004		0.0088	0.0028	0.0074
Zr	0.002		0.0025	0.0026	

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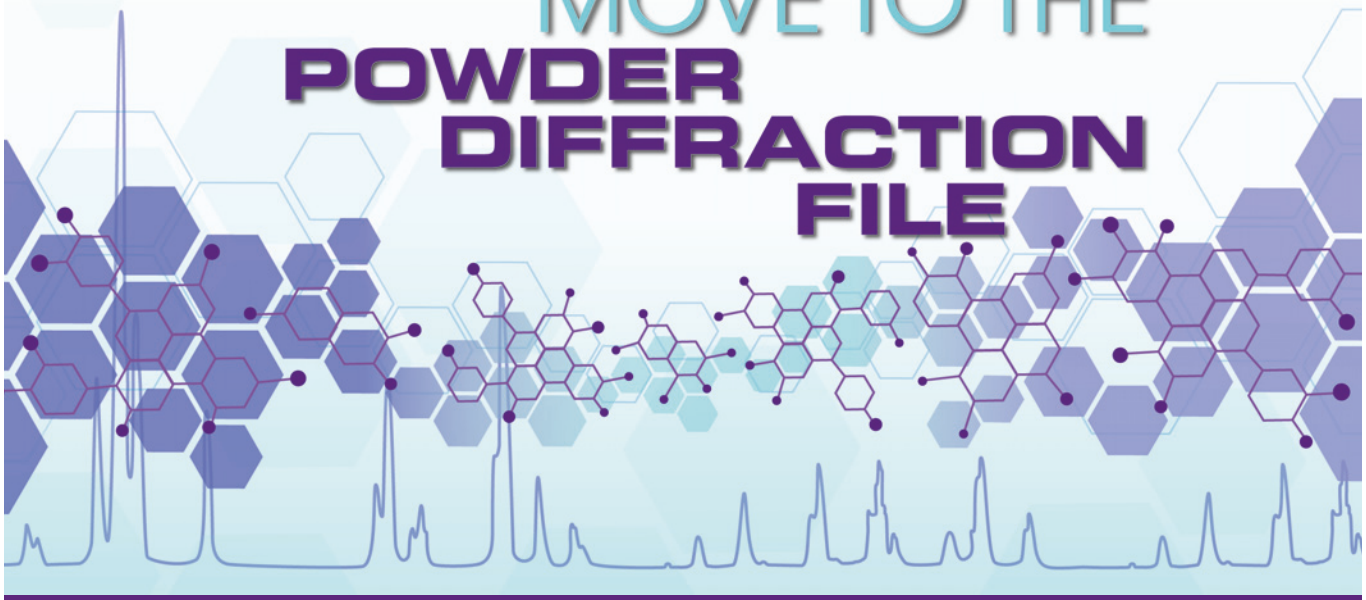

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Data Entry Source	PDF-2 2016	PDF-4+ 2016 WebPDF-4+ 2016	PDF-4/ Minerals 2016	PDF-4/ Organics 2017
00- ICDD	115,066	115,066	12,031	39,395
01- FIZ	165,264	67,498	12,294	12,185
02- CCDC	0	0	0	431,359
03- NIST	10,067	2,960	216	281
04- MPDS	0	198,367	19,748	0
05- ICDD Crystal Data	722	722	52	32,785
Total No. of Data Sets	**291,119**	**384,613**	**44,341**	**516,054**
Subfile Distribution:				
Inorganic	258,130	351,612	44,285	33,943
Organic	41,386	42,229	701	505,530
New Entries	12,616	18,736	1,489	14,090
Rietveld—No. with atomic coordinates	0	271,449	33,574	96,825
Reference Intensity Ratio (RIR)—I/I_c	193,567	286,885	33,362	484,021
Experimental Digital Patterns	0	11,287	127	6,062
Pattern Fitting—Calculated Digital Patterns	0	384,613	44,341	516,054

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PRESS RELEASE

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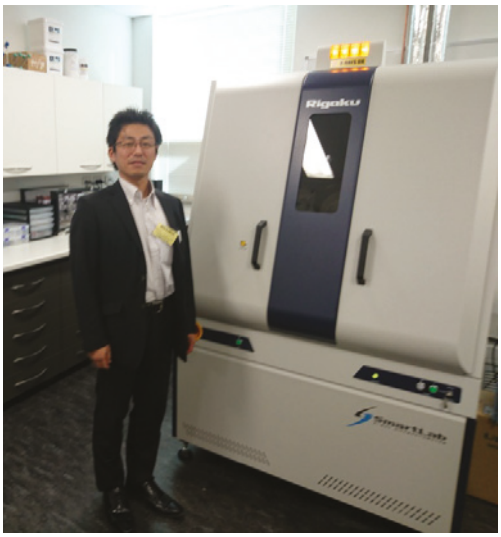
CSIRO Enhance Minerals Analysis Capabilities with 9kW X-Ray Diffractometer

Sydney, Australia, August 1, 2016 - AXT is proud to announce that they have recently commissioned another 9kW Rigaku SmartLab X-Ray Diffractometer (XRD). This is the third high-flux XRD to have been installed in Australia and demonstrates that Australia has embraced the technology and advantages that it brings. This system has been installed at CSIRO's Waite Campus in Adelaide.



The Rigaku SmartLab is arguably one of the world's most versatile XRD systems. Its range of capabilities can be increased markedly with a host of attachments that can also be added at a later date, including third party attachments. These enable a wider range of measurements to be performed under a variety of configurations and non-ambient conditions. They can all be controlled using Rigaku's user-friendly guidance software, with Rigaku's attachment being optically encoded so the system automatically checks to ensure they are correctly installed.

CSIRO have optioned this system with an automatic sample changer which will aid unattended analyses, capillary spinner for analysis of fibres and plate-like crystals as well as transmission stage, SAXS (Small Angle X-ray Scattering) and USAXS (Ultra Small Angle X-Ray Scattering) for characterisation of clays, colloids and other nano-particulates. They also have an Anton Paar CHC+, controlled atmosphere chamber for *in-situ* monitoring of crystallographic changes at temperatures between -5°C and 350°C in air or other gasses (and up to 400°C under vacuum) and in humid atmospheres between 5% and 95% RH at temperatures from 10°C to 60°C so they can determine how environmental and processing variables will affect materials.

CSIRO's Mineralogical and Geochemical Characterisation Team Leader Mark Raven uses the 9 kW Rigaku XRD system to assist with his research. Mark said, "our main area of research is characterising geological and mineral samples specialising in characterising clays and clay minerals. The ability to work in SAXS and USAXS modes as well as controlling temperature and humidity is crucial for our work and these were the key features we required."

Mark and his colleagues' expertise has been recognised with a win in 2010 and second placing in this year's Reynold's Cup quantitative mineralogy round robin. This global competition pits commercial, government, industrial and academic laboratories against one another. They are charged with producing the most accurate quantitative results from supplied mixtures of pure standards that represent realistic sedimentary rock compositions.

AXT represents Rigaku in Australia and New Zealand distributing their WDXRF, single crystal and protein crystallography systems as well as computed tomography and radiography equipment. For more information on their product lines and other scientific characterisation instruments from a host of international suppliers, please visit www.axt.com.au.

AXT Install Australia's First High Speed Simultix 14 XRF Spectrometer

Sydney, Australia, May 5 2016 - AXT has recently installed the first Rigaku Simultix 14 high-speed Wavelength Dispersive X-Ray Fluorescence spectrometer (WDXRF) at BHP Billiton's nickel operation. This system is now up and running 24/7 characterising ore samples to supporting their mining operation.

The Simultix 14 is a high-throughput, high-precision system for elemental analysis, suited to measuring light to heavy elements from beryllium to uranium. It is a highly advanced system that features full automation and has been designed to meet the most stringent precision and throughput laboratory requirements while maintaining analytical flexibility and versatility. BHP have optioned their system with the 48 position auto sample changer to increase its ability to run unattended.

Rigaku have engineered in many smart features that have resulted in a fully automated, high performance instrument with a relatively small footprint. These features include state-of-the-art high frequency circuits, highly efficient heat exchanger, faster vacuum system and high speed sample loading system.

These performance features make the Simultix 14 an ideal process control instrument that can be easily integrated into your workflow to improve quality and throughput.

TRhe system is also extremely flexible with a maximum of 40 channels. Other options include automated background correction and sequential light and heavy element goniometer.

The Simultix 14 is the latest evolution of the Simultix family of multi-channel simultaneous XRF analysers, benefitting from more than 30 years of experience. This high-performance system has been designed to meet the increasing needs of industries such as steel and cement manufacturing.

Speaking about this particular installation, AXT's Managing Director Richard Trett said, "while installing this high-end XRF system, the first in Australia is a great achievement, it is more pleasing to know that Australian mining is still alive and willing to invest in solutions like the Simultix 14 to maintain quality and efficiency. We hope to work with more Australian miners to improve quality control for their operations."

AXT represent Rigaku in Australia selling their WDXRF, XRD, CT and NDT product lines. They also have agencies for numerous other synergistic manufacturers around the world enabling them to offer tailored solutions for industry and academia. For more details visit www.axt.com.au.

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